JC20 Rec'd PCT/PTO SEP 2 0 2001

09/937124

JORDAN AND HAMBURG LLP
122 East 42nd Street,
New York, NY 10168
Tel: (212) 986-2340 Fax: (212) 953-7733
Customer No.: **000028107**

Docket No.: **F-7160**
Filing Date: **September 20, 2001**

Certificate of Express Mailing Under 37 CFR 1.10

"Express Mail" label number: EL666236198 US Date of Deposit: 9/20/01

I hereby certify that this correspondence is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 in an envelope addressed to ASSISTANT COMMISSIONER OF PATENTS, WASHINGTON, DC 20231 having the above "Express Mail" label number on the date of deposit indicated above.

Madeline Gonzalez	Madeline Gonzalez
(Typed or printed name of person mailing paper or fee)	(Signature of person mailing paper or fee)

10937124 . 092001

THE ASSISTANT COMMISSIONER FOR PATENTS
Washington, D. C. 20231

[ ] ATTN: BOX PATENT APPLICATION

[ ] ATTN: BOX DESIGN PATENT APPLICATION

[x] ATTN: BOX PCT

[x] THIS IS THE 35 U.S.C 371 NATIONAL STAGE OF **PCT/DE00/00747** FILED **March 9, 2000**

Sir:

Transmitted herewith for filing is the [x] Utility [ ] Design nonprovisional patent application of:

Inventor / Application Identifier: **Birgit SEIDEL, et al.**

[x] See Inventor Information Sheet attached

For: **METHOD FOR CONTROLLING CRYSTAL SIZE DURING CONTINUOUS MASS CRYSTALLISATION**

[ ] This is a new patent application.
[x] This is the 35 U.S.C. 371 National Stage Application of the above-identified PCT Application.
[ ] This is a: [ ] Continuation Application
[ ] Divisional Application
[ ] Continuation-in-Part Application
of prior Application Serial No. _.

[ ] Cancel in this application original claims ___ of the prior application before calculating the filing fee.

[ ] Amend the specification by inserting before the first line the sentence:
-- This is a [ ] Continuation, [ ] Division, [ ] Continuation-in-part, of Application

[ ] Incorporation By Reference. The entire disclosure of the prior application, from which a copy of the oath or declaration is supplied, is considered as being part of the disclosure of the accompanying application and is hereby incorporated by reference therein.

09/937124

20 SEP 2001

ENCLOSED ARE THE FOLLOWING:		
X	3	Sheets of drawings ([x] formal [ ] informal size A4).
X	11	Pages of specification including abstract and claims.
X	14	Total pages.
	Combined Declaration and Power of Attorney	
		Newly executed
		Copy from prior application
		Inventors deleted; see attached statement
	Sequence Listing	
		Computer Readable Copy
		Paper copy
		Statement verifying identity of above copies
X	Return Receipt Postcard	
	Preliminary Amendment	
	Assignment to:	
		Assignment is of record in prior application Serial No._.
		Assignment Recordation Form Cover Sheet.
		Charge $40.00 to Deposit Account No. 10-1250 for recording Assignment.
X	Information Disclosure Statement	
X	Information Disclosure Citation	
	English translation	
X	Application Data Sheet	

PRIORITY CLAIMS		
	Applicant hereby claims the benefit of the filing date of the following provisional application(s) under the provision of 35 USC 119.	
X	Applicant hereby claims the benefit under the provisions of 35 USC 119 of the filing dates of the following applications as indicated below: **Germany Patent Appln. No. 199 12 699.2, filed March 20, 1999, Priority Claimed** of which certified copies thereof	
		will follow
		are enclosed
	X	have been filed in the International Bureau
		were filed in prior application:

CLAIMS FILED AND FILING FEE CALCULATION					
ITEM	—			Rate	Applied Fee
[ ] Base Fee - Non PCT	—			$710	
[ ] Base Fee - PCT IPEA-US	—			$690	
[ ] Base Fee - PCT ISA-US	—			$710	
[ ] Base Fee - PCT not ISA or IPEA	—			$1,000	
[X] Base Fee - PCT with EPO or JPO Search Report	—			$860	$860
[ ] Base Fee - Design	—			$320	
Claim Fees	Number Filed	Base Number	Number Extra over Base	—	
Total Claims	0	20	0	$18	$0
Independent Claims	0	3	0	$80	$0
Multiple Dependent Claim Fee	—			$270	$0
[X] Small Entity Status is Asserted	—				($430)
[X] Foreign Language Filing Fee	—			$130	$130
TOTAL FILING FEE					**$560**

09/937124

JC03 Rec'd PCT/PTO 20 SEP 2001

[X] Please charge Deposit Account No. 10-1250 in the amount of the above TOTAL FILING FEE. A duplicate copy of this sheet is attached.

[X] Please charge to Deposit Account No. 10-1250 any further fees due for filing or during prosecution of this application under: 37 CFR 1.16; 37 CFR 1.17; and 37 CFR 1.492.

JORDAN AND HAMBURG LLP

By____________________

C. Bruce Hamburg
Reg. No. 22389
Attorney for Applicant

10/22/01 4:21:24 PM

09/937124
JC03 Rec'd PCT/PTO 20 SEP 2001

INVENTOR INFORMATION SHEET

Docket Number: F-7160
Title: METHOD FOR CONTROLLING CRYSTAL SIZE DURING CONTINUOUS MASS CRYSTALLISATION
Filing Date: 9/20/01

1. Full Name of Inventor **Birgit SEIDEL**	Family Name **SEIDEL**	First Given Name **Birgit**	Second Given Name
Citzenship **Germany**	Residence City **Luppenau**	Residence State or Province	Residence Country **Germany**
Postal Address **An der Kastanienallee 5**	City **Luppenau**	Province/State	Postal Code/Country **D-06254 Germany**
2. Full Name of Inventor **Peter ULLRICH**	Family Name **ULLRICH**	First Given Name **Peter**	Second Given Name
Citzenship **Germany**	Residence City **Zwintschoena**	Residence State or Province	Residence Country **Germany**
Postal Address **Eschengrund 18**	City **Zwintschoena**	Province/State	Postal Code/Country **D-06184 Germany**
3. Full Name of Inventor **Michael ZEIBIG**	Family Name **ZEIBIG**	First Given Name **Michael**	Second Given Name
Citzenship	Residence City **Spergau**	Residence State or Province	Residence Country **Germany**
Postal Address **Strasse der OdF 6**	City **Spergau**	Province/State	Postal Code/Country **D-06237 Germany**
4. Full Name of Inventor **Dieter SCHMITT**	Family Name **SCHMITT**	First Given Name **Dieter**	Second Given Name
Citzenship **Germany**	Residence City **Leuna**	Residence State or Province	Residence Country **Germany**
Postal Address **Sachsenstrasse 2**	City **Leuna**	Province/State	Postal Code/Country **D-06237 Germany**

10/937124.092601

09/937124

Application Data Sheet

Application Information

Application Type::	Regular
Subject Matter::	Utility
Suggested Group Art Unit::	
Sequence submission?::	
Computer Readable Form (CRF)?::	
Title::	METHOD FOR CONTROLLING CRYSTAL SIZE DURING CONTINUOUS MASS CRYSTALLISATION
Attorney Docket Number::	F-7160
Suggested Drawing Figure::	1
Total Drawing Sheets::	3
Small Entity::	Yes

Applicant Information

Applicant Authority Type::	Inventor
Primary Citizenship Country::	Germany
Status::	Full Capacity
Given Name::	Birgit
Middle Name::	
Family Name::	SEIDEL

09/937124

20 SEP 2001

City of Residence::	Luppenau
State or Province of Residence::	
Country of Residence::	Germany
Street of Mailing Address::	An der Kastanienallee 5
City of Mailing Address::	Luppenau
State or Province of Mailing Address::	
Country of Mailing Address::	Germany
Postal or Zip Code of Mailing Address::	D-06254
Applicant Authority Type::	Inventor
Primary Citizenship Country::	Germany
Status::	Full Capacity
Given Name::	Peter
Middle Name::	
Family Name::	ULLRICH
City of Residence::	Zwintschoena
State or Province of Residence::	
Country of Residence::	Germany

10937124.092001

09/937124

20 SEP 2001

Street of Mailing Address:: Eschengrund 18

City of Mailing Address:: Zwintschoena

State or Province of Mailing Address::

Country of Mailing Address:: Germany

Postal or Zip Code of Mailing Address:: D-06184

Applicant Authority Type:: Inventor

Primary Citizenship Country::

Status:: Full Capacity

Given Name:: Michael

Middle Name::

Family Name:: ZEIBIG

City of Residence:: Spergau

State or Province of Residence::

Country of Residence:: Germany

Street of Mailing Address:: Strasse der OdF 6

City of Mailing Address:: Spergau

State or Province of Mailing Address::

Country of Mailing Address:: Germany

10937124 .092201

Postal or Zip Code of Mailing Address:: D-06237

Applicant Authority Type:: Inventor

Primary Citizenship Country:: Germany

Status:: Full Capacity

Given Name:: Dieter

Middle Name::

Family Name:: SCHMITT

City of Residence:: Leuna

State or Province of Residence::

Country of Residence:: Germany

Street of Mailing Address:: Sachsenstrasse 2

City of Mailing Address:: Leuna

State or Province of Mailing Address::

Country of Mailing Address:: Germany

Postal or Zip Code of Mailing Address:: D-06237

Correspondence Information

Correspondence Customer Number:: 000028107

Representative Information

Representative Designation::	Registration number::	Name::
Primary	22389	C. Bruce Hamburg

Domestic Priority Information

Application::	Continuity Type::	Parent Application::	Parent Filing Date::
This application	National Stage of	PCT/DE00/00747	03/09/00

Foreign Priority Information

Country::	Application Number::	Filing Date::	Priority Claimed::
Germany	199 12 699.2	03/20/99	Yes

F-7160

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant	:	Birgit SEIDEL et al.
Serial No.	:	09/937,124
For	:	METHOD FOR CONTROLLING CRYSTAL SIZE DURING CONTINUOUS MASS CRYSTALLISATION
Group Art Unit	:	Not yet known
Examiner	:	Not yet known

Assistant Commissioner for Patents
Washington, D.C. 20231

PRELIMINARY AMENDMENT

Sir:

Preliminary to examination, please amend this application as follows:

IN THE CLAIMS:

Cancel claim 1.

Add the following claims 7-11:

--7. In a continuous mass crystallization conducted in a crystallization medium in a crystallizer, a method for controlling the size of crystals during the mass crystallization, comprising producing a seeding product independently of the mass crystallization, the average particle diameter of solids of the seeding product

being 0.1 to 1.0 mm and smaller than crystalline material produced by the mass crystallization, and introducing the seeding product into the crystallizer while maintaining temperature thereof up to 40°C lower than the temperature of the crystallization medium, all other materials fed or recycled into the crystallizer being free of solids.

8. A method according to claim 7, wherein the crystallization is of ammonium sulfate.

9. A method according to claim 7 or 8, wherein said temperature of the seeding product is 10 to 30°C lower than the temperature of the crystallization medium.

10. A method according to claim 3, wherein the amount of the seeding product introduced into the crystallizer based on the solids discharged from the crystallizer is 7 to 15% by weight.

11. A method according to claim 7 or 8, wherein the solids of the seeding product are produced by a separate crystallization.--

Amend claims 2-6 as follows, the amendments being shown by brackets and underlining in the Appendix hereto:

2. (Amended) A method according to claim 7, wherein the seeding product is introduced into the crystallizer discontinuously in such a manner that the proportion by weight of a selected fraction of the crystalline material in the crystallizer is maintained within predetermined limits.

3. (Amended) A method according to claim 7, wherein the seeding product is introduced into the crystallizer continuously and the solids of the seeding product are introduced into the crystallizer in amounts of 5 to 30% by weight based on solids discharged from the crystallizer.

4. (Amended) A method according to claim 7 or 8, wherein the average particle diameter of the solids of the seeding product is 0.3 to 0.8 mm.

5. (Amended) A method according to claim 7 or 8, wherein the solids of the seeding product are produced by mechanical communition of crystals produced by the mass crystallization.

6. (Amended) A method according to claim 7, wherein the solids of the seeding product have the same chemical composition as the crystals produced by the mass crystallization.

REMARKS

This places the application in better condition for examination by presenting claims suitable for U.S. practice.

Attached hereto on a separate page is an Abstract to be added as the last page of the specification.

Please charge the $140 multiple dependent claim fee to Deposit Account No. 10-1250. Also charge any fee deficiency or credit any overpayment to the same deposit account.

Respectfully submitted,

Jordan and Hamburg LLP

By C. Bruce Hamburg
C. Bruce Hamburg
Reg. No. 22,389
Attorney for Applicants

and,

By Frank J. Jordan
Frank J. Jordan
Reg. No. 20,456

Jordan and Hamburg LLP
122 East 42nd Street
New York, New York 10168
(212) 986-2340

Enclosure: Abstract

APPENDIX I

AMENDED CLAIMS WITH AMENDMENTS INDICATED THEREIN BY BRACKETS AND UNDERLINING

2. (Amended) A method [for controlling the size of the crystals during the continuous mass crystallization of] according to claim [1] 7, wherein[, for discontinuous seeding,] the seeding product is [added] introduced into the crystallizer discontinuously in such a manner[,] that the proportion by weight of a selected fraction of the crystalline material in the crystallizer [remains] is maintained within [specified] predetermined limits.

3. (Amended) A method [for controlling the size of the crystals during the continuous mass crystallization of] according to claim [1] 7, wherein [during continuous] the seeding[,] product is introduced into the crystallizer continuously and the solids [portion] of the seeding product [is added] are introduced into the crystallizer in amounts of 5 to 30% by weight [and preferably of 7 to 15% by weight,] based on [the] solids discharged from the crystallizer.

4. (Amended) A method [for controlling the size of the crystals during the continuous mass crystallization of one or more of claims 1 to 3] according to claim 7 or 8, wherein the average particle diameter of the solids of the seeding product is 0.3 to 0.8 mm.

5. (Amended) A method [for controlling the size of the crystals during the continuous mass crystallization of one or more of the claims 1 to 4] according to claim 7 or 8, wherein [the desired particle size of] the solids of the seeding product [is] are produced by mechanical communition of crystals produced by the [end product and/or in a separated] mass crystallization [step].

6. (Amended) A method [for controlling the size of the crystals during the continuous mass crystallization of one or more of the claims 1 to 5]according to claim 7, wherein the solids of the seeding product [has] have the same chemical composition as the [end product] crystals produced by the mass crystallization.

ABSTRACT

The invention relates to a method for controlling the crystal size during continuous mass crystallisation, especially with ammonium sulphate. According to said method, seed products are added, the seed product being produced independently of the current crystallisation process in terms of its parameters. The average grain size of the solid form of the seed product is 0.1 to 1.0 mm and is smaller than that of the desired crystallisate. The solid form of the seed product is produced from different technological sub-streams in the given frain size range, independenly of the main crystallisation process. The temperature of the seed product when it is added is up to 40°C, preferably 10 to 30°C lower than the process temperature in the crystalliser and all other materials that are supplied or returned to the crystalliser are free of solids. By controlling the parameters of the seed product it is possible to influence the grain size distribution of the end product and significantly reduce the fluctuations in the distribution of the grain size of the end product. The method can be carried out with discontinuous or continuous addition of the seed product.

METHOD FOR CONTROLLING THE SIZE OF CRYSTALS DURING CONTINUOUS MASS CRYSTALLIZATION

During mass production by crystallization, the particle size must comply with strict specifications.

In order to minimize the manufacturing costs of such products, it is necessary to come as close as possible to these particle size distributions already during the crystallization process and to produce these distributions stably. The invention therefore relates to a method for controlling the size of crystals during a continuous mass crystallization.

Especially ammonium sulfate, as fertilizer or industrial product, is produced preferably by crystallization processes. For fertilizer production, a coarsely crystalline product with a defined particle size spectrum is demanded, in order to guarantee the required scatter and scatter accuracies. The industrial products should be of a finer crystalline size.

The mode of functioning and construction of a draft tube buffer crystallization apparatus (DTB crystallizer) are known (US patent 3,873,275)..

With this, the required particle size distributions can be produced, but cannot be produced stably. Because of their constructive design from the point of view of minimizing the formation of fine particles by the selective destruction of crystallization nuclei, the particle size distribution, especially of crystals produced in DTB crystallizers, has a great tendency to fluctuate cyclically.

An apparatus with a dynamic control method is also known. For this, various process variable, such as the rate of recycling the fine crystals, the flow of feed solution, the pH, the degree of mixing or the supply of seeding crystals is controlled on the basis of the analysis of the particle size distribution of the crystalline material in the crystallizing apparatus and, with that, a uniform particle size distribution is obtained (US patent 4,263,010).

However, this method is technologically very expensive and can hardly be designed stably.

A method for producing large crystals with a DTB crystallizer is also known. The offtake of crystals from the DTB crystallizer (production rate) varies depending on the determined density of the suspension in the crystallizing apparatus, the power consumption of the stirrer motor, the height of the crystalline bed under the baffle and the size distribution of the crystals (JP 150 127).

Admittedly, the proportion of crystals larger than 1.4 mm is increased. However, the proportion of crystals larger than 2.0 mm still fluctuates between 35% and 90%. The alternating, fluctuating production rate and, with that, the inadequate utilization of the installed capacity of the plant are extremely serious disadvantages for the downstream industrial units.

Furthermore, a method is known, by means of which the proportion of larger crystals in a DTB crystallizer is increased. For this method, a suspension of crystals with 6% to 25% by volume of crystals is supplied to the crystallizer, the solids of this suspension constituting 4% to 25% by weight of the solids withdrawn from the crystallizer. For this method, 35% to 85% by weight of the seeding crystals are larger than 1.2 mm and not more than 15% by weight of the crystals are larger than 1.7 mm (WO 93/19826).

The temperature of the seeding suspension is lower than the temperature of the crystallizer.

It is a disadvantage of the invention that only an averaging of the production and an increase in the proportion of larger crystals is achieved. Neither the selective control of the size of the crystals nor the production of a product with finer crystals is described or claimed.

It was therefore an object of the invention to eliminate these disadvantages, that is, to find a method for reproducibly controlling the size of the crystals during a continuous mass crystallization.

Pursuant to the invention, this is accomplished by a method, in which seeding products are added

- the seeding product, in its parameters, being produced independently of the actual crystallization process,
- the average particle diameter of the solids of the seeding products being 0.1 to 1.0 mm and smaller than that of the desired crystalline material,
- the solids of the seeding product being produced independently of the main process of crystallization from different industrial partial flows in the specified particle size region
- the temperature of the seeding product during the addition being as much as 40°C and preferably 10° to 30°C lower than the process temperature in the crystallizer and
- all other materials, fed and recycled into the crystallizer, being free solids.

A suspension of crystals, the parameters of which can be adjusted completely independently of the actual crystallization process, is supplied to the crystallization apparatus. This suspension is characterized by the solids content, by

the particle size distribution and by the amount of product supplied to the crystallization apparatus in unit time.

The particle size distribution of the final product is affected by controlling the parameters of this seeding product and the fluctuations in the particle size distribution of the end product (solids taken off from the crystallization apparatus), are reduced.

The precise parameters of the seeding product for a given crystallization apparatus can be obtained empirically in relation to the desired, steady state of this apparatus.

This method can be carried out by adding the seeding product discontinuously as well as continuously.

When seeding discontinuously, the seeding product is added discontinuously in such a manner, that the proportion by weight of a selected fraction of the crystalline material in the crystallizer remains with in specified limits.

To prevent strong, cyclic fluctuations in the size of the crystals of the end product, an effective seed formation rate is required, which is adequate for the system, fluctuates slightly and is reflected in constant proportions over time of the individual fractions, particularly the fractions less than 1.0 mm. When the limiting range is not attained, seeding is carried out and, when the limiting range is exceeded, the seeding is suspended.

When seeding continuously, the proportion of solids of the seeding product is added in amounts of 5 to 30 percent by weight and preferably of 7 to 15 percent by weight, based on the solids, discharged, the crystallizer.

Advisably, the average particle diameter of the solids of the seeding product is 0.3 to 0.8 mm. In any case, it is less than the particle diameter of the desired, crystalline material.

The desired particle size of the solids of the seeding product can be adjusted by known procedures. Preferably, it is produced by a mechanical comminution of one of the fractions of the end product and/or in a separate stage of the crystallization. In other words, the end product is not used unchanged.

The seeding product need not be the same chemical substance as the end product of the continuous mass crystallization. However, it is advantageous if the seeding product has the same chemical composition as the end product. For example, crystals of ammonium sulfate are used for seeding during the continuous mass crystallization of ammonium sulfate.

The advantages of the invention lie

- in the defined control of the particle size distribution and, with that, of the average particle diameter of the end product,
- in the prevention of excessive cyclic fluctuations in the particle size distribution of the end product and, with that, in an improved utilization of the plant capacity,
- therein that the actual crystallization process has no effect on the control parameter (on the particle size of the seeding product) and
- that, during continuous seeding, the number of screen analyses for controlling the process can be decreased drastically.

Figure 1 shows a flow chart of the inventive, continuous, mass crystallization. A key for the symbols is given below:

1. crystallization apparatus (crystallizer)
2. pipeline (for feed solution)
3. pipeline (for vapors)
4. vapor compressor
5. heat exchanger
6. circulating pump
7. circulating pipeline
8. pipeline (for mash)
9. mash pump
10. centrifuge
11. interim storage tank
12. pump
13. pipeline (for mother liquor)
14. pipeline (for crystalline material)
15. pipeline (full partial flow)
16. elutriation crystallizer
17. centrifuge
18. pipeline (for crystalline material or part of the seeding product)
19. tank
20. pipeline for part of the seeding product
21. metering and conveying system
22. pipeline for seeding product

The crystallization is carried out in a continuous crystallization apparatus (1), preferably in an OSLO or a DRAFT TUBE BAFFLE (DTB) crystallizer.

The pre-heated feed solution (for example, with 37 $\pm$ 3 percent by weight of ammonium sulfate) is passed through pipeline (2) into the crystallizer.

The vapors arising are aspirated over pipeline (3) by a vapor compressor (4) and are compressed. The energy of the compressed vapors is transferred by means of heat exchangers (5) and a circulating pump (6) through the circulating pipeline (7) into the crystallization equipment.

Mash is withdrawn continuously through pipeline (8) and supplied with a mash pump (9) to a centrifuge (10). The mother liquor, which has been separated off, reaches an interim storage tank (11) and is transferred with the pump (12) over the pipeline (13) into the circulating pipeline (7). Over the pipeline (14), the crystalline material reaches the downstream processing plant.

A partial flow (liquid phase) is removed over pipeline (15) from the crystallization equipment and transferred to the elutriation crystallizer (16). The crystalline material, obtained here, is separated by a centrifuge (17) and added over pipeline (18) to the tank (19) as a possible component of the so-called seeding product.

A partial flow of the seeding product (for example, of ammonium sulfate crystals), which was produced, in relation to particle size distribution and amount by mechanical comminution of a partial amount of the end product, is also supplied to the tank (19) over the pipeline (20). From this, a pumpable suspension of crystals is produced and supplied, with the help of a metering and conveying system (21), over the pipeline (22) to the crystallizer in such a manner, that the crystals of the seeding product cannot settle.

The invention is described by the following examples, without being limited to these.

<u>Example 1</u> (Comparison Example Without Additional Seeding Product)

In a continuously operating crystallization apparatus (DTB crystallizer), the active portion is about 280 m^3.

The preheated feed solution (ammonium sulfate solution, 38.5 $\pm$ 2% by weight ammonium sulfate content and a temperature of about 90°C) is supplied to the crystallizer without additional seeding product. The evaporation rate is 30T/H and the production rate is 20T/H (crystals withdrawn from the crystallizer). The solids content of the mash in the crystallizer is 35 to 40% by weight.

The particle size distribution, measured by means of the fraction greater than 1.8 mm over a period of 120 hours, is shown in Figure 2. The fluctuations in the particle size distributions are very large.

<u>Example 2</u> (discontinues addition of seeding product)

The basic operating state corresponds to that of Example 1, with the exception of the explicit requirement that, aside from the seeding product, all other materials supplied and recycled to the crystallization equipment must be absolutely free of solids.

By means of a particle size analysis of the crystalline particles from the interior of the crystallization equipment, a fraction is selected, the size range of which should be close to the average diameter of the particles of the seeding product.

The mass flow of seeding product is controlled discontinuously by means of defined upper and lower limiting values of this fraction, which can be determined empirically. The fraction greater than 0.4 mm and less than 1.0 mm of the crystalline material in the crystallization apparatus is used for the start and end of the discontinuous seeding. When this fraction falls below 1% by weight, the

crystallization apparatus is seeded. The solids portion of the seeding product is 10% by weight and the average particle diameter is about 0.6 mm..

When the fraction selected exceeds 2% by weight, seeding is suspended. If the upper limiting value is clearly exceeded, under some circumstances in conjunction with changes in other operating parameters, the specified parameter can be restored by supplying plant condensate to the crystallization apparatus.

The particle size distribution, measure by means of the fraction greater 1.8 mm over a period of 120 hours, is shown in Figure 3.

<u>Example 3</u> (continues addition of seeding product)

The basic operating state corresponds to that of Example 1, however, with the explicit requirement that, aside from the seeding product, anything else, supplied to or recycled into the crystallization apparatus, must be absolutely free of solids.

The seeding product is supplied continuously to the crystallization equipment with fixed parameters, which are optimized empirically. The solids content of the seeding product is 7% by weight, based on the end product that is discharged and the average particle diameter is 0.6 mm. The seeding product is added continuously at the rate of 15 m^3/h.

Particle size analyses as a basis for controlling the operating state, are no longer required or can, at the very least, be reduced clearly in number. The particle size distribution, measured by means of the fraction of greater than 1.8 mm over a period of 120 hours, is shown in Figure 4.

Example 4 (continues addition of seeding product)

The operating state corresponds to that of Example 3.

The seeding product is supplied continuously to the crystallization apparatus with fixed parameters, which are optimized empirically.

The solids content of the seeding product is 25% by weight, based on the discharged end product, and the average particle diameter is about 0.6 mm. The seeding product is metered in continuously at a rate of 25 m^3/h. The particle size is reduced selectively by an excess of seeding product.

The particle size distribution, measure by means of the fraction, greater than 1.8 mm over a period of 120 hours, is shown in Figure 5. In the period between the 24^{th} and the 80^{th} hour, the fraction greater than 1.8 mm is selectively moved into the 20% range by seeding.

Claims

1. A method for controlling the size of the crystals during continuous mass crystallization, especially of ammonium sulfate, by the addition of seeding products, wherein

- the seeding product, in its parameters, is produced independently of the actual crystallization process,
- the average particle diameter of the solids of the seeding products is 0.1 to 1.0 mm and smaller than that of the desired crystalline material,
- the solids of the seeding product are produced independently of the main process of crystallization from different industrial partial flows in the specified particle size range,
- the temperature of the seeding product during the addition is as much as 40°C and preferably 10° to 30°C lower than the process temperature in the crystallizer and
- all other materials, fed and recycled into the crystallizer, are free solids.

2. A method for controlling the size of the crystals during the continuous mass crystallization of claim 1, wherein, for discontinuous seeding, the seeding product is added discontinuously in such a manner, that the proportion by weight of a selected fraction of the crystalline material in the crystallizer remains within specified limits.

3. A method for controlling the size of the crystals during the continuous mass crystallization of claim 1, wherein during continuous seeding, the solids portion of the seeding product is added in amounts of 5 to 30% by weight and preferably of 7.to 15% by weight, based on the solids discharged from the crystallizer.

4. A method for controlling the size of the crystals during the continuous mass crystallization of one or more of claims 1 to 3 wherein the average particle diameter of the solids of the seeding product is 0.3 to 0.8 mm.

5. A method for controlling the size of the crystals during the continuous mass crystallization of one or more of the claims 1 to 4, wherein the desired particle size of the solids of the seeding product is produced by mechanical communication of the end product and/or in a separated crystallization step.

6. A method for controlling the size of the crystals during the continuous mass crystallization of one or more of the claims 1 to 5, wherein the seeding product has the same chemical composition as the end product.

COMBINED DECLARATION FOR PATENT APPLICATION A
POWER OF ATTORNEY
(Includes Reference to PCT International Applications)

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

METHOD FOR CONTROLLING CRYSTAL SIZE DURING CONTINUOUS MASS CRYSTALLISATION

the specification of which (check only one item below):

[ ] is attached hereto.

[ ] was filed as United States application

Serial No. ________________

on ________________,

and was amended

on ________________ (if applicable).

[X] was filed as PCT international application

Number PCT/DE00/00747

on March 9, 2000

and was amended under PCT Article 19

on ________________ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §119(a)-(d) or (f), §365(b) of any foreign application(s) for patent or inventor's certificate or of any PCT international application(s) designating at least one country other than the United States of America listed below and have also identified below any foreign application(s) for patent or inventor's certificate or any PCT international application(s) designating at least one country other than the United States of America filed by me on the same subject matter having a filing date before that of the application(s) of which priority is claimed:

PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 U.S.C. 119:			
Country (if PCT indicate "PCT")	Application Number	Date of Filing	Priority Claimed Under 35 USC 119
Germany	199 12 699.2	March 20, 1999	[X] Yes [ ] No

COMBINED DECLARATION FOR PATENT APPLICATION AND POWER OF ATTORNEY (Continued) (Includes Reference to PCT International Applications)	Attorney's Docket Number F-7160

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) or PCT international application(s) designating the United States of America that is/are listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in that/those prior application(s) in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application(s) and the national or PCT international filing date of this application:

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

C. Bruce Hamburg	Reg. No. 22389
Frank J. Jordan	Reg. No. 20456
Herbert F. Ruschmann	Reg. No. 35341
Jacqueline M. Steady	Reg. No. 44354
Derek S. Jessen	Reg. No. 48213

(5)

Send Correspondence To:	Jordan ~~and Hamburg~~ LLP 122 ~~East 42nd Street~~ ~~New York, New~~ York 10168	Direct Telephone Calls to: C. Bruce Hamburg (212) 986-2340

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Full Name of First or sole Inventor	Inventor's Signature	Date
Birgit SEIDEL	Birgit Seidel	10.10.01
Residence An der Kastanienallee 5 D-06254 Luppenau, Germany		Citizenship Germany
Post Office Address An der Kastanienallee 5 D-06254 Luppenau, Germany DEX		

Full Name of Second Inventor	Inventor's Signature	Date
Peter ULLRICH	Peter [illegible]	09.10.2001
Residence Eschengrund 18 D-06184 Zwintschoena, Germany		Citizenship Germany
Post Office Address Eschengrund 18 D-06184 Zwintschoena, Germany DEX		

Full Name of Third Inventor	Inventor's Signature	Date
Michael ZEIBIG		09.10.01
Residence Strasse der OdF 6 D-06237 Spergau, Germany		Citizenship Germany
Post Office Address Strasse der OdF 6 D-06237 Spergau, Germany		

Full Name of Fourth Inventor	Inventor's Signature	Date
Dieter SCHMITT		09.10.2001
Residence Sachsenstrasse 2 D-06237 Leuna, Germany		Citizenship Germany
Post Office Address Sachsenstrasse 2 D-06237 Leuna, Germany		

United States Patent & Trademark Office

Office of Initial Patent Examination -- Scanning Division

Application deficiencies found during scanning:

☑ Page(s)__________ of Drawing__________ were not present for scanning.
(Document title)

☐ Page(s)__________ of____________________ were not present for scanning.
(Document title)

☐ ***Scanned copy is best available.***